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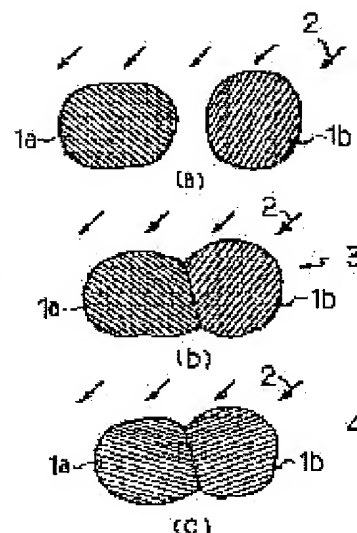
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(54) PRODUCTION OF FUSED ULTRAFINE PARTICLES

(57)Abstract:

PROBLEM TO BE SOLVED: To enable each of ultrafine particles to be fused to each other under controlled conditions and increase the stabilization of fused ultrafine particles which are important in the application of fused ultrafine particles.

SOLUTION: At least two ultrafine metallic particles adjacent to each other 1a, 1b are irradiated with high-energy beams 2, for example, electron beams to fuse the two ultrafine metallic particles thereby forming a fused ultrafine particle 3 having a corresponding grain boundary. These fused ultrafine metallic particles 3 are further irradiated with high-energy beams to lower the Σ value in the corresponding grain boundaries thereby producing stabilized ultrafine fused metallic particles 4.



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CLAIMS

[Claim(s)]

[Claim 1]A manufacturing method of ultrafine particle fusant characterized by comprising the following:

it adjoins --- at least ---, irradiating two ultra-fine particles with a high energy beam --- said --- at least --- A process of generating ultra-fine particle fusant which unites two ultra-fine particles and has a coincidence boundary

A process which irradiates further with a high energy beam ultra-fine particle fusant which has said coincidence boundary, and stabilizes said ultra-fine particle fusant.

[Claim 2]A manufacturing method of ultrafine particle fusant reducing sigma value of a coincidence boundary of said ultra-fine particle fusant by said stabilization process in a manufacturing method of the ultrafine particle fusant according to claim 1.

[Claim 3]A manufacturing method of ultrafine particle fusant which ultra-fine particle fusant to which it is said stabilization process and sigma value of said coincidence boundary was further reduced in a manufacturing method of the ultrafine particle fusant according to claim 2 is made into a single crystal grain or a polycrystal grain, and is characterized by decreasing a defect within said single crystal grain or a polycrystal grain.

[Claim 4]A manufacturing method of ultrafine particle fusant characterized by comprising the following:

To two or more ultra-fine particles distributed on a substrate, it irradiates with a high energy beam simultaneously, and adjoins. A process of uniting said two or more ultra-fine particles, and generating two or more ultra-fine particle fusants

A process which said two or more ultra-fine particle fusants are further irradiated with a high energy beam, and sigma value of ultra-fine particle fusant which has a coincidence boundary among said two or more ultra-fine particle fusants is reduced, and is stabilized.

[Claim 5]A manufacturing method of ultrafine particle fusant both characterized by a thing which are said stabilization processes and makes said two or more ultra-fine particle fusants further a single crystal grain or a polycrystal grain in a manufacturing method of the ultrafine particle fusant according to claim 4, and for which a defect within said single crystal grain or a polycrystal grain is decreased.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacturing method of ultra-fine particle fusan.

[0002]

[Description of the Prior Art] In metal particles or a compound particle, it is the particle diameter. If it ultrafine-particle—size like 100 nm or less, the different characteristic from the usual particles (for example, 1 micrometers or more) will appear. Since such an ultrafine particle can apply the characteristic which is suitable for discovery of a new surface phenomenon, control of the outline, etc., and is different in the usual bulk etc. to various fields, research on the physical properties of the ultrafine particle itself, application of an ultrafine particle, etc. is advanced.

[0003] Conventionally, the ultrafine particle is produced by a physical method and the chemical method as shown below. Namely, as a manufacturing method of a physical ultrafine particle, Gas evaporation which evaporates metal etc. in inactive gas, makes it cool and condense by the collision with gas, and generates an ultrafine particle. The metallo-fumes synthetic method which heats metal under the sputtering process which uses a weld slag phenomenon as an evaporation source, and a vacuum, carries out vapor codeposition of the metal atom which evaporated on the substrate cooled below to the coagulating point of the organic solvent with the organic solvent, and obtains an ultrafine particle, the flow oil top vacuum evaporation process which makes metal vapor—deposit on oil, etc. are mentioned.

[0004] As a manufacturing method of the chemical ultrafine particle using the liquid phase, The colloid method which returns a precious-metals salt under flowing-back conditions in the alcohol which made the polymer surfactant live together. The alkoxide process using hydrolysis of a metal alkoxide, the coprecipitation method which adds a precipitant to the mixed liquor of metal salt, and obtains precipitate particles, etc., As a manufacturing method of the chemical ultrafine particle which furthermore used the gaseous phase, A thermal decomposition method of the organo metallo compound which obtains an ultra-fine particle by thermal decomposition reactions, such as a metal carbonyl compound, Metal chloride is heated in a reactant gas air current, reduction, oxidation, and nitriding of the metal chloride which and obtains an ultrafine particle, and an oxide and a hydrous ghost are heated in a hydrogen air current, and the reduction in hydrogen to return, the solvent evaporation method to which hot air drying of the metal salt solution is sprayed and carried out from a nozzle, etc. are mentioned. [metal chloride] [oxidize or]

[0005]

[Problem(s) to be Solved by the Invention] By the way, the conventional research and development to an ultrafine particle are related mainly with the aggregate (superfines object) of an ultrafine particle, and it cannot be said that research on the various operations and control to an ultrafine particle simple substance is fully done. It is because this was difficult to originate also in the manufacturing method of the conventional ultrafine particle mentioned above, and to obtain an ultrafine particle as a particle simple substance in the conventional manufacturing method. Since it is such, it will not have resulted, by the time it aims at application deployment by making an ultrafine particle simple substance into the charge of a start material.

[0006] For example, although becoming overly producible [detailed output various device, and various function material etc.] by realizing under the conditions by which fusion of ultrafine particles, etc.

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were controlled is predicted. Since research on fusion control of ultrafine particles, etc. is not fully done, it will not have resulted, by the time it aims at application deployment which was described above.

[0007] Since it is such, the art which made it possible to unite under the conditions by which ultrafine particle simple substances were controlled is searched for, and when applying the fusan of ultrafine particles further, stabilization technique etc. of the ultrafine particle fusan which becomes important are desired.

[0008] This invention was made in order to enable application deployment from an ultrafine particle simple substance, etc., for example, and an object of this invention is to enable fusion of ultrafine particle simple substances under the controlled conditions, and to provide the manufacturing method of the ultrafine particle fusan which realized stabilization of ultrafine particle fusan.

[0009]

[Means for Solving the Problem] This invention is characterized by a manufacturing method of ultrafine particle fusan comprising the following.

irradiating at least two adjoining ultra-fine particles with a high energy beam — said — at least — . Process of generating ultra-fine particle fusan which unites two ultra-fine particles and has a coincidence boundary

A process which irradiates further with a high energy beam ultra-fine particle fusan which has said coincidence boundary, and stabilizes said ultra-fine particle fusan.

[0010] A manufacturing method of ultrafine particle fusan of above-mentioned this invention, It is characterized by reducing sigma value of a coincidence boundary of said ultra-fine particle fusan especially by said stabilization process, and by said stabilization process. Ultra-fine particle fusan to which sigma value of said coincidence boundary was furthermore reduced is made into a single crystal grain or a polycrystal grain, and it is characterized by decreasing a defect within said single crystal grain or a polycrystal grain.

[0011] This invention is characterized by a manufacturing method of other ultrafine particle fusants comprising the following.

To two or more ultra-fine particles distributed on a substrate, it irradiates with a high energy beam simultaneously, and adjoins. Process of uniting said two or more ultra-fine particles, and generating two or more ultra-fine particle fusants

A process which said two or more ultra-fine particle fusants are further irradiated with a high energy beam, and sigma value of ultra-fine particle fusan which has a coincidence boundary among said two or more ultra-fine particle fusants is reduced, and is stabilized.

[0012] A manufacturing method of ultrafine particle fusan of above-mentioned this invention makes further ultra-fine particle fusan to which sigma value of said coincidence boundary was further reduced by said stabilization process a single crystal grain or a polycrystal grain, and it is characterized by decreasing a defect within said single crystal grain or a polycrystal grain.

[0013] That is, this invention adjoins — at least — By irradiating two ultra-fine particles with a high energy beam, By being able to unite these ultra-fine particles and irradiating further ultra-fine particle fusan which has the obtained coincidence boundary with a high energy beam, Based on having found out, it accomplishes that it is possible in reducing sigma value of a coincidence boundary in a fusion interface of ultra-fine particle fusan a single crystal grain or to be able to carry out polycrystal granulation and to decrease a defect within a single crystal grain or a polycrystal grain in ultra-fine particle fusan

[0014]

[Embodiment of the Invention] Hereafter, the gestalt for carrying out this invention is explained.

[0015] Drawing 1 is a figure showing typically one embodiment of the manufacturing process of the ultrafine particle fusan by this invention. first, as shown in drawing 1 (a), it adjoins — at least — The two ultra-fine particles 1a and 1b are irradiated with the high energy beam 2.

[0016] As the ultra-fine particle 1, the ultra-fine particle which forms coincidence boundaries, such as Pt ultrafine particle, Au ultrafine particle, and Cu ultrafine particle, is mentioned here, and it is the particle diameter. It is preferred that it is about 2-30 nm. If the particle diameter of the ultra-fine particle 1 as a starting material exceeds 30 nm, there will be a possibility that a fusion phenomenon

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cannot be induced even if it irradiates with the high energy beam 2, and particle diameter The production itself is difficult for the ultra-fine particle 1 below 2 nm. It adjoins. As for the interval of the two ultra-fine particles 1a and 1b, it is preferred that it is a particle diameter grade. If operation and control are possible for the ultra-fine particle 1 used as a starting material which was described above as an ultra-fine particle simple substance, a manufacturing method in particular is not limited and can use what was obtained by the manufacturing method of various ultra-fine particles.

[0017] Especially the high energy beam 2 with which the ultra-fine particle 1 is irradiated should just have not the thing limited but the energy which may unite the two ultra-fine particles 1a and 1b. For example, intensity The electron beam more than $1 \times 10^{19} \text{ e/cm}^2$ and sec, a corpuscular beam like the ion beam which has intensity equivalent to this electron beam, a photon like laser, X-rays, a gamma ray, a neutron beam, etc. are mentioned.

[0018] When using an electron beam as the high energy beam 2, irradiation intensity There is a possibility that the ultra-fine particle 1 may not be activable, so that ultra-fine particle fusant can be generated as it is less than $1 \times 10^{18} \text{ e/cm}^2$ and sec. In other words, the electron beam (2) which has the intensity more than $1 \times 10^{18} \text{ e/cm}^2$ and sec brings about an activation effect, the partial heating effect, etc. of the ultra-fine particle 1, and becomes generable [ultra-fine particle fusant] by these. The intensity of an electron beam is practical. It is preferred that it is more than $1 \times 10^{19} \text{ e/cm}^2$ and sec. The same may be said of the case where a corpuscular beam, a photon, X-rays, a gamma ray, a neutron beam, etc. are used as the high energy beam 2.

[0019] What is necessary is just to set up according to a use beam, and a vacuum atmosphere, an inert atmosphere like argon atmosphere, etc. may be mentioned, and an oxygen containing atmosphere, a nitrogen atmosphere, etc. may be sufficient as the exposure atmosphere of the high energy beam 2 depending on the case. For example, atmosphere in the case of applying electron beam irradiation It is preferred to consider it as the vacuum atmosphere below $1 \times 10^{-3} \text{ Pa}$, and it can prevent adsorption of a residual gas atom etc. by this. The exposure of the high energy beam 5 can be carried out on a room temperature stage.

[0020] It adjoins. If it irradiates with the high energy beam 2 which was described above to the two ultra-fine particles 1a and 1b simultaneously, the ultra-fine particles 1a and 1b will be activated, and it will begin to approach, and as finally shown in drawing 1 (b), the ultra-fine particle 1a and 1b will unite. Since it is random, here the crystal orientation of the ultra-fine particles 1a and 1b before irradiating with the high energy beam 2 in the ultra-fine particle fusant (it is hereafter described as initial fusant) 3 of the time of fusion. Although based on the crystal orientation of the ultra-fine particles 1a and 1b before an exposure, distance, the intensity of the high energy beam 2, etc., sigma value of the coincidence boundary in a fusion interface of large probability is high. It can be said that the initial fusant 3 with large sigma value of such a coincidence boundary has large surface energy, and is in an unstable state.

[0021] The above-mentioned sigma value has a crystal structure and an equal lattice constant. It is an index which shows in what kind of direction two crystals are generating the periodical structure of what kind of interval by facing across the grain boundary. That is, if they assume that it has a rotation relation around a specific rational crystal axis supposing the penetration lattice of both crystals when the state where it has touched by the interface with two crystals is considered, when taking a specific value with an angle of rotation, some lattice points of both crystals lap. When the lattice which this overlapping position makes is called a correspondence lattice (Coincidence Site Lattice: CSL) and a lattice point is in agreement at a rate of $1/\text{sigma}$ among the lattice points of both crystals, that CSL is displayed with sigma value (always odd number). The simplest correspondence lattice except the same crystal in cubic system is sigma 3. It is a grain boundary.

[0022] If it irradiates with the high energy beam 2 further to the initial fusant 3 which was described above, the atomic arrangement of each ultra-fine particles 1a and 1b will change, and sigma value of the coincidence boundary of the initial fusant 3 will fall so that surface energy may decrease in a fusion interface. that is, the further exposure of the high energy beam 2 shows the initial fusant 3 to drawing 1 (c) — as — sigma value of a coincidence boundary — for example, — 3 — as — it becomes the small ultra-fine particle fusant 4. This ultra-fine particle fusant 4 can be said to be the ultra-fine particle fusant (it is hereafter described as metastable-sized fusant) which has the stable coincidence boundary from it being in the most stable state (sigma value is the minimum) in the

fusant which has a coincidence boundary.

[0023] If it irradiates with the high energy beam 2 further to the above-mentioned metastable-sized fusant 4, the atomic arrangement of each ultra-fine particles 1a and 1b will change, for example, it will become a single crystal grain so that the crystal face and crystal orientation in a fusion interface may become the same. Under the present circumstances, although lattice defects, subgrain boundaries, etc., such as a rearrangement, disclination, and a point defect, usually exist in a crystal grain, these lattice defects and a subgrain boundary are also decreased or extinguished by change of atomic arrangement. Thus, the exposure of the high energy beam 2 to the metastable-sized fusant 4 brings about stabilization of ultra-fine particle fusant, such as reduction or disappearance of a lattice defect or a subgrain boundary in single-crystal-grain-izing of fusion particles, and a single crystal grain.

[0024] The change to the metastable-sized fusant 4 mentioned above from the initial fusant 3, and the change from the metastable-sized fusant 4 to a single crystal grain etc. Since [such as intensity of the high energy beam 2, irradiation time, original crystal orientation, distance of the ultra-fine particles 1a and 1b with which it irradiates,] it therefore differs, a fixed state is not necessarily acquired always, for example, may become a polycrystal grain, but, A more stable state is realizable with sufficient reproducibility, so that irradiation time is so long that the intensity of the high energy beam 2 is large. Since it was such, when an electron beam is used for the intensity of the high energy beam 2, for example It is preferred to use more than $1 \times 10^{19} \text{ e/cm}^2$ and sec, and it is irradiation time. It is preferred to consider it as 100 seconds or more.

[0025] Thus, it adjoins — at least — By irradiating the two ultra-fine particles 1a and 1b with the high energy beam 2, By being able to obtain the ultra-fine particle fusant (initial fusant) 3, and irradiating this initial fusant 3 with the high energy beam 2 further, The fusant of ultra-fine particles can be stabilized, such as reducing sigma value of the coincidence boundary in a fusion interface, or decreasing a lattice defect, a subgrain boundary, etc., after single-crystal-grain-izing fusion particles further. Therefore, when overly applying to detailed output, various device, and various function material etc., for example, it becomes possible to provide the ultra-fine particle fusant stabilized in various states.

[0026] In the above-mentioned embodiment, the fusant of the ultra-fine particles which have not passed through the molten state, and the further stable ultra-fine particle fusant can be formed on a room temperature stage. Since it is difficult to irradiate with high energy beams, such as an electron beam, under the controlled heating conditions generally, enabling generation of the ultra-fine particle fusant on a room temperature stage has a large meaning.

[0027] By the way, it adjoins in drawing 1. Although only the two ultra-fine particles 1a and 1b were illustrated, the ultra-fine particle as a starting material of ultra-fine particle fusant may be the ultra-fine particle 1 of a large number distributed on the substrate 5, as shown, for example in drawing 2. In such a case, if many ultra-fine particles 1 are simultaneously irradiated with the high energy beam 2, it adjoins. The two or more ultra-fine particles 1 and 1 — unite, and two or more ultra-fine particle fusants generate.

[0028] Although sigma value of the ultra-fine particle fusant which has a coincidence boundary among such two or more ultra-fine particle fusants changes with the crystal orientation of the ultra-fine particle 1 before irradiating with the high energy beam 2, distance, the intensity of the high energy beam 2, etc., For example, sigma 3 with sigma value of a coincidence boundary small according to the irradiation time of the high energy beam 2 The ultra-fine particle fusant which has a grain boundary increases. That is, when it sees as two or more whole ultra-fine particle fusants, stabilization of ultra-fine particle fusant progresses.

[0029] The increase in small, stabilization of two or more above-mentioned ultra-fine particle fusants, i.e., sigma value of a coincidence boundary, fusant is realizable in a short time, so that the intensity of the high energy beam 2 with which it irradiates is large. The number of ultra-fine particle fusants with small sigma value of a coincidence boundary increases, and it can stabilize by the case where it sees as two or more whole ultra-fine particle fusants, so that the intensity of the high energy beam 2 is large, when in other words the irradiation time of the high energy beam 2 is the same.

[0030] If it irradiates with the high energy beam 2 further to two or more ultra-fine particle fusants which were [above-mentioned] stable, single-crystal-grain-izing of each fusant particle, the further fusion of two or more fusant particles, etc. will take place, and the metal super-thin film in which a

single crystal grain and a polycrystal grain are intermingled will be obtained. Under the present circumstances, like the embodiment mentioned above, a lattice defect, a subgrain boundary, etc. in a crystal grain are decreased or extinguished, and will be in a more stable state.

[0033] Thus, when it sees as two or more whole ultra-fine particle fusants by irradiating many ultra-fine particles 1 with the high energy beam 2 simultaneously, ultra-fine particle fusant can be stabilized and the metal super-thin film in which a single crystal grain and a polycrystal grain are intermingled further can be obtained.

[0032]

[Example] Next, the concrete example of this invention is described.

[0033] Example 1 — the example of production of Pt ultrafine particle used for manufacture of ultrafine particle fusant is described first. That is, as shown in drawing 3, the carbon film 12 has been arranged as a substrate on the support member 11, and the Pt mesh 13 (thickness: 200 micrometers) which serves as a starting material of Pt ultrafine particle on this carbon film 12 has been arranged.

This Pt mesh 13 is a target material, and a diameter. It has much 100-micrometer fine pores 14.

[0034] These are set on the room temperature stage in a vacuum chamber, and it is in the Pt mesh 13 about the carbon film 12. It is from an oblique direction to the fine-pores wall 14a of the Pt mesh 13 about the accelerating voltage of 3.0 kV, and the Ar ion beam 15 of 0.25 mA of beam current, making it rotate at 2 rpm. It glared for 180 seconds. The incidence angle theta of the Ar ion beam 15 was 40 degrees. Atmosphere at the time of Ar ion beam irradiation was made into the vacuum about 1×10^{-3} Pa (Ar is included).

[0035] When TEM observation of the carbon film 12 top was carried out after the exposure of the above-mentioned Ar ion beam 15, it was checked that the Pt ultrafine particle 16 is formed in the position corresponding to the fine pores 14 of the Pt mesh 13 on the carbon film 12, respectively. The TEM observation result is typically shown in drawing 4. As shown in drawing 4, after Pt ultrafine particle had dissociated on the carbon film, a large number existed. Diameter of these Pt(s) ultrafine particle is about 5 nm. The electron diffraction pattern of Pt ultrafine particle immediately after production is typically shown in drawing 5. Drawing 5 shows that Pt ultrafine particles immediately after formation are low crystalline particles.

[0036] Thus, Pt ultrafine particle of obtained a large number is received — inside of the vacuum atmosphere of 1×10^{-3} Pa it irradiated with the electron beam of 1.0×10^{20} e/cm² and sec. It is the above-mentioned electron beam to drawing 6. The electron diffraction pattern of Pt ultrafine particle of the stage with which it irradiated for 240 seconds is shown typically. Drawing 6 shows that the crystallinity of Pt ultrafine particle is improving.

[0037] It is the above-mentioned electron beam to drawing 7. The TEM observation result after glaring for 700 seconds is shown typically. Drawing 7 shows that Pt ultrafine particles unite by the exposure of an electron beam, and Pt ultrafine particle fusant is generating. Drawing 8 is a figure showing typically the HETRM image of the field enclosed with the solid line of drawing 7. To Pt ultrafine particle adjacently arranged from drawing 8 Electron beam of 1.0×10^{20} e/cm² and sec By glaring for 700 seconds, [many] It turns out that Pt ultrafine particle fusant which has a coincidence boundary of sigma 3 (111), a coincidence boundary of sigma 11 (113), and a coincidence boundary of sigma 19 (331) is obtained, respectively. In this stage, there are many rates of Pt ultrafine particle fusant with still large sigma value of a coincidence boundary like sigma 19, and when it sees as the whole Pt ultrafine particle fusant, it cannot be said that it fully stabilizes.

[0038] Then, it is an electron beam of the same conditions further to Pt ultrafine particle fusant of the state which showed in drawing 7 and drawing 8. When it glared for 300 seconds, sigma value of a coincidence boundary is small, for example, it is sigma 3. Pt ultrafine particle fusant which has a grain boundary increased. Thus, Pt ultrafine particle fusant can be stabilized by irradiating Pt ultrafine particle fusant with an electron beam further.

[0039] As opposed to Pt ultrafine particle of a large number obtained like the above-mentioned example on the other hand — electron beam of 2.1×10^{20} e/cm² and sec It glared for 700 seconds. The TEM observation result after this electron beam irradiation is typically shown in drawing 9. It compares, when it irradiates with the electron beam of 1.0 from drawing 9, 1×10^{20} e/cm² and sec, and it is more sigma 3. It turns out that Pt ultrafine particle fusant which has a grain boundary (an arrow shows among a figure) is obtained. Thus, stabilization of Pt ultrafine particle fusant can be attained in

a short time by enlarging intensity of the electron beam with which Pt ultrafine particle is irradiated. [0040] As opposed to Pt ultrafine particle of a large number obtained like example 2 Example 1 — inside of the vacuum atmosphere of 1×10^{-3} Pa It irradiated with the electron beam of 3.3×10^{20} e/cm² and sec. To drawing 10, it is an electron beam about the mimetic diagram of the TEM observation result in front of electron beam irradiation. The mimetic diagram of the TEM observation result after a 1000-second exposure is shown for the mimetic diagram of the TEM observation result of the stage with which it irradiated for 300 seconds in drawing 11 figure 12 again.

[0041] Drawing 10 and drawing 11 show that fusion of Pt ultrafine particles takes place by the exposure of an electron beam. By irradiating with an electron beam further, sigma value of a coincidence boundary is small, for example, it is sigma 3. It was also checked that Pt ultrafine particle fusant which has a grain boundary increases. To drawing 13, it is the above-mentioned Example 1. In two electron beam intensity and the electron beam intensity of Example 2, it is an electron beam. The ratio of the ultrafine particle fusant which has each sigma value after glaring for 700 seconds is compared and shown. By enlarging intensity of the electron beam with which Pt ultrafine particle is irradiated from drawing 13, sigma value of a coincidence boundary is small, for example, it is sigma 3. More Pt ultrafine particle fusants which have a grain boundary are obtained, and it turns out that stabilization of Pt ultrafine particle fusant is realizable in a short time.

[0042] From drawing 12, after performing generation of fusant, and the fall of sigma value, by continuing irradiating with an electron beam further shows obtaining the Pt super-thin film in which a single crystal grain and a polycrystal grain are intermingled. The above-mentioned electron beam From the TEM observation result of the stage with which it irradiated for 700 seconds, it was checked that there will be few rearrangements in a crystal grain, and the Burgers vector will almost be $a/3 \langle 11\bar{2} \rangle$, a rearrangement etc. arise by electron beam irradiation further, and a defect decreases.

[0043]

[Effect of the Invention] As explained above, according to the manufacturing method of the ultrafine particle fusant of this invention, it can obtain with sufficient reproducibility under the conditions by which the ultrafine particle fusant which is excellent in stability, such as a fusion interface, was controlled. Thus, since stability, such as a fusion interface, is controllable according to this invention, it contributes to the application deployment from an ultra-fine particle simple substance, etc. greatly.

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- The drawings, any words are not translated.

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Ultra-fine particle fusant (initial fusant)

100